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# PATENT SPECIFICATION



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## COMPLETE SPECIFICATION

### Process for the Production of Titanium

We, FARBENFABRIKEN BAYER AKTIENGESELLSCHAFT, of Leverkusen-Bayerwerk, Germany, a Body Corporate organised under the Laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The present invention relates to a process for the production of titanium.

It is known that titanium halides can be reduced with alkali or alkaline-earth metals in order to obtain titanium. The reaction is 15 generally carried out by placing the alkali or alkaline-earth metal in a reaction vessel, heating to the reaction temperature of 700-900°C. and then introducing the titanium halide, using argon as a protective gas. The 20 titanium which is formed separates out in the form of a sponge. Moreover, during the reduction, there is also formed alkali metal halide or alkaline-earth metal halide which is present in the form of a melt at the reaction temperature and is run off through suitable devices after the reaction is completed. The halide which remains contained in the titanium sponge in addition to excess reduction metal is to a large extent distilled off 30 by heating the reaction vessel under vacuum. After this treatment, the titanium sponge is so firmly set in the reaction vessel that it has to be removed with the aid of mechanical devices such as milling devices or drills. 35 It has now been found that the reduction of titanium halide can be carried out technically in a substantially more simple manner if the reduction metal, i.e. alkali or alkaline-earth metal or metals, is used in the form of an alloy with zinc, cadmium or lead or a mixture of two or all of these metals, which alloy is liquid at the reaction temperature, and if the gaseous titanium halide is introduced into this liquid alloy. 45 The term alkaline-earth metal as used

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herein includes magnesium. The separated titanium is then suspended in the liquid alkali metal or alkaline-earth metal halide melt being simultaneously formed, settles satisfactorily in the said melt and can be 50 pumped off from the boundary layer between the liquid alloying metal and the alkali metal or alkaline-earth metal halide melt.

All titanium halides, but preferably 55 titanium chloride, can be used for the present process. The amount thereof which is added to the reduction metal is preferably such that the latter remains in excess in order to avoid reformation of titanium chloride in the alkali or alkaline-earth metal 60 chloride melt.

The process may be carried out, for example as follows:

The reduction metal, for example magnesium, is dissolved in liquid zinc, cadmium, lead or a mixture thereof and the gaseous titanium halide is thereafter introduced at the reaction temperature, which is in the region of 600°C. The introduction of the said 70 halide can be carried out with all technical devices known for such purposes. One preferred method consists in that the gaseous titanium chloride is forced into the metal melt through a bottom plate which is per- 75 meable to gas.

The separated titanium is suspended in the alkali or alkaline-earth metal chloride melt being simultaneously formed in the reaction, settles therein and may be pumped 80 off from the boundary layer between the alloying metal and the clarified alkali or alkaline-earth chloride melt. The alloy is advantageously covered from the outset with the corresponding chloride, for example car- 85 nalite.

The further working up process may consist in allowing the titanium-containing suspension which is pumped off to settle and then filtering off the titanium. The filter cake 90

containing titanium may be freed from the excess melt and the reduction and alloying metals, by heating the said cake, for example in a vacuum furnace heated by radiant heat.

5 The pure titanium sponge can be melted down in the usual manner.

One particular advantage of the process according to the invention consists in that the process may also be carried into effect 10 continuously. In this case, the alloy, consisting of the reduction metal and the alloying metal, is preferably prepared externally of the reaction vessel and the liquid alloy is caused to flow continuously through the 15 reaction chamber. The alloy, which issues from the reaction chamber and of which the content of reduction metal is to a large extent consumed, can be re-introduced into the cycle again after adding fresh reduction 20 metal.

The circulation of the alloy may either be effected with the aid of induction currents, which at the same time serve for heating purposes, or by mechanical means similar 25 to those known in connection with the circulation of metallic mercury in alkali metal chloride electrolysis cells. The liquid cathode metal in this case preferably runs over weirs, so that the electrolysis sludge is retained in 30 the cell, from which it may be extracted from time to time.

In one preferred form of the process according to the invention the reduction metal is produced in the required amount directly 35 within the alloy metal by fusion electrolysis, using the liquid alloy metal as cathode. The electrolyte used may consist of alkaline-earth or alkali metal chlorides or mixtures thereof or other fusion electrolytes, which can be 40 electrolysed to yield the alkaline-earth or alkali metal with which the titanium compounds are to be reduced.

The titanium suspension formed in the melt of the alkaline-earth or alkali metal 45 chlorides may also be pumped off continuously or intermittently, and can be worked up according to the process indicated above.

An additional advantage of the present process lies in the fact that the heat of re- 50 action liberated during the reduction may be dissipated by suitable cooling means either during circulation of the alloy or of the melt. By this means, it is possible to produce a high reaction velocity. At the same 55 time, it is also possible in a simple manner to control and regulate the maintenance of the required excess of reduction metal with respect to the titanium halides.

If necessary, the process can also be carried 60 into effect with exclusion of air or in an inert gas atmosphere.

#### EXAMPLE 1.

2000 gm. of cadmium are melted and 52 gm. of magnesium are added to this melt. 65 The metal melt is heated to 600°C. and

covered with a carnallite melt. Thereafter, titanium tetrachloride is introduced through the porous base of the vessel. During the reaction, there are formed 203 gm. of magnesium chloride and about 52 gm. of titanium metal in powder form, which settles in the carnallite melt and is removed as a suspension. The titanium suspension is filtered, the filter cake is liberated from excess melt and alloy metal in a vacuum furnace 75 which is provided with radiation heating and the titanium which is obtained is melted down in the usual manner in an arc furnace.

#### EXAMPLE 2.

A sodium chloride-calcium chloride melt 80 containing about 25% of NaCl and 75% of CaCl<sub>2</sub> is electrolysed in a suitable electrolysis cell resulting in the hourly production of 90-100 gm. of sodium and corresponding equivalent of calcium in the liquid 85 cathode which consists of cadmium metal. The cathode metal flows over weirs into a reaction vessel, in which the reduction metal is reacted with gaseous titanium tetrachloride, and then flows back into the cell. The 90 temperature in the electrolysis cell is 600-650°C. 190 gm. of gaseous titanium tetrachloride per hour are introduced into the reaction vessel through a gas permeable base plate. The amount of titanium tetrachloride 95 corresponds approximately to the amount of reduction metal which is hourly produced. When it enters the reaction vessel, the cathode metal contains about 5% of the reduction metal, and about 0.5 to 1%, thereof 100 on leaving the said vessel. In the reaction vessel, 48 gm. of titanium separate hourly as titanium powder and 240 gm. of common salt or corresponding calcium chloride equivalents are produced in the same period. The 105 current density in the electrolysis cell is 28,000-30,000 amp./m<sup>2</sup>, and the current efficiency 90-95%. The titanium suspension which separates in the reaction vessel in the alkaline-earth metal or alkali metal 110 chloride melt is extracted by vacuum from time to time from the reaction vessel, allowed to settle and filtered. The filter cake containing titanium is fed to a radiation furnace operated with vacuum. In this furnace, 115 the remaining melt and reduction metals are distilled off. The titanium which is obtained is thereafter melted down in an arc furnace in the usual manner. All operations are carried out with exclusion of air. 120

#### EXAMPLE 3.

The procedure followed in this example is in principle the same as that described in Example 2. Zinc is used instead of cadmium as the alloy metal and magnesium is electro- 125 lysed into the zinc from a carnallite melt. 52 gm. of magnesium are separated out every hour and this is reacted in the reaction cell with the theoretical amount of titanium tetrachloride. 204 gm. of magnesium chlo- 130

ride and 51 gm. of titanium metal are formed each hour. The temperature in the electrolysis cell is 700-750°C. As in Example 2, the operation is carried out with an excess of magnesium metal which remains in the cycle.

What we claim is:—

1. A process for the production of titanium from titanium halide by reduction with alkaline-earth or alkali metals, wherein at least one alkaline-earth or alkali metal is dissolved in zinc, cadmium or lead or a mixture of two or all of these metals to form a liquid alloy and the titanium halide is introduced into this liquid alloy.
- 15 2. A process as claimed in Claim 1, wherein the liquid alloy is covered with an alkaline-earth or alkali metal chloride melt, from which the titanium formed is pumped off.
3. A process as claimed in Claim 1 or 2, wherein the process is carried out continuously.

4. A process as claimed in any of Claims 1 to 3, wherein an excess of alkali or alkaline-earth metal is used in relation to the titanium halide.

5. A process as claimed in Claim 1, wherein the alkali or alkaline-earth metal is directly deposited in the alloying metal by fusion electrolysis.

6. A process for the production of titanium from titanium halide substantially as described with reference to any of the Examples.

7. Titanium whenever produced by the process claimed in any of the preceding claims.

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